THE CRYSTAL STRUCTURE OF POTASSIUM HYDROGEN FUMARATE, $KC_4H_3O_4$

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The potassium salts of fumaric acid have been studied by Gupta (1956), Gupta and Barnes (1961). The present work reports the preliminary results of X-ray crystal structure analysis of potassium hydrogen fumarate, $KC_4H_3O_4$. The crystallographic data for the substance are given below and are the same as reported by Gupta and Barnes (ibid)

$a = 6.952$Å, $b = 7.483$Å, $c = 6.24$Å, $\alpha = 107° 05'$, $\beta = 117° 00'$

$\gamma = 96° 04'$, $V = 265.5A^3$, $\rho = 1.936$ gm/cm^3

$Z = 2$; Sp.gr.$P\bar{1}$; μ linear absorption co-efficient for CuKα = 82.0 cm^{-1}

Reflexions of the type *okl*, *hol*, *hko*, *hl̄l* and *hkh̄* were collected using small single crystals and Weissenberg normal beam zero layer photography around appropriate crystallographic axes. Intensities were estimated visually. There was some difficulty initially in locating the potassium atoms from the Patterson projections but once they were located, the heavy atom technique was adopted and after normal Fourier methods had been exhausted, least squares refinement of the experimental data was undertaken, using only an overall isotopic temperature factor and unit weights. Figure below gives a view of the structure as looking down the [100] axis.

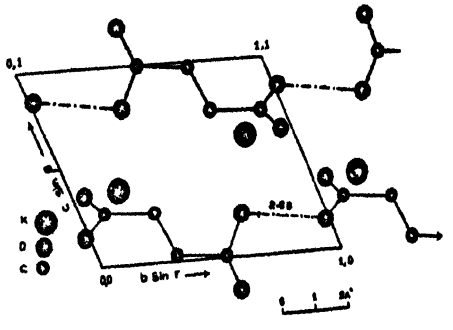

Fig. 1

R factors after several cycles of least squares refinements are

R(*okl*) 14.5; *R*(*hol*) 14.0; *R*(*hko*) 16.5; *R*(*hl̄l*) 16.2;

The bond lengths and angles of the fumarate group are similar to those reported for fumaric acid (Brown 1966, Post *et al*, 1966). The interesting features of the structure, however, are as follows :

(1) The fumarate group, unlike in the fumaric acid structure, is non-planar, one of the two carboxyl groups being twisted by as much as 35° out of the plane of the rest of the atoms.

(2) Hydrogen bonding of 2.63Å between two adjacent molecules, forming an extensive chain of molecules inside the crystal. The hydrogen bonds, moreover, are between two oxygen atoms, both of which are of the type OH...OH (i.e. longer of the two C-O bonds in a COOH group) and this situation is similar to that observed in the structure of bisphenylacetate, $(C_6H_5CH_2COO)_2HK$, (Speakmanl 949, Bacon and Curry 1957).

(3) A five-fold co-ordination of oxygen atoms around the metal ion with K^+—O distances ranging from 2.74Å to 3.03Å. The surrounding of the metal ions by the oxygen atoms provides for combinations of distorted octahedra and tetrahedra.

As the result (1) and (2) above have interesting points of stereochemistry and packing to settle, further work is being done to refine the structure using complete three dimensional data. As this may take some time, we are publishing here the essential features of this crystal structure and we believe that these results are not likely to be modified to any marked extent even after a full three dimensional analysis.

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COSMIC RAY FLUXES AT DIFFERENT ZENITH ANGLES

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An experiment has been performed to find out the variation of integral cosmic-ray fluxes with zenith angles. The geographical co-ordinates at the place of measurement are Lat. 22° 34'N, Long. 88° 24'E and height from sea-level is 20 ft.

A four-fold Geiger-Müller counter telescope has been utilized for this purpose and the fluxes have been recorded by a four-fold coincidence circuit. The counter telescope has been placed under a thin Aluminium foil of 0,06 mm thickness.